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Science Highlights

from the National Synchrotron Light Source

Observation of Nitrogen Polarization in Fe-N Using Soft X-ray Magnetic Circular Dichroism

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Fe-nitrides together with transition metal oxides are part of a new generation of interesting magnetic materials extensively investigated because of their potential use in future magnetic devices. Fe-N is a complex system, with a rather complicated phase diagram containing multiple phases like γ'-Fe_4N, ε'-Fe_3N, ξ-Fe_2N and α"$Fe_{16}N_2$. The α" $Fe_{16}N_2$ phase is the most interesting phase, with a reported magnetic moment up to 4 times that of the Fe one. The fabrication of single-phase samples of Fe-N is currently a complicated task, especially for thin films of the α" phase. Our interest to study Fe-N thin films is driven by the search for a better understanding of nitrogen's role in the magnetic properties of this system. Previous structural reports place the N atoms in an interstitial position inside the Fe structure, suggesting rather weak interactions between Fe and N.

BEAMLINE
X13A

PUBLICATION
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Using x-ray Magnetic Circular Dichroism (MCD), we studied a series of Fe-N thin films in a thickness range between 15 and 100 nm with predominantly γ'-Fe_4N phase. Prior to the MCD measurements, the samples were structurally characterized with x-ray diffraction (XRD) and magnetically characterized with a Superconducting Quantum Interference Device (SQUID), making use of the Polar Magneto-Kerr effect (MOKE) **(Figure 1)**. The P-MOKE data show an expected 4-fold symmetry as the Fe-N thin films were grown on a (001) FCC MgO substrate shown by the XRD measurements.

MCD measurements were collected at the National Synchrotron Light Source beamline X13A. This beamline delivers modulated soft x-rays in an energy range between 200 and 1600 eV, switching between left and right polarization with frequencies close to 22 Hz. MCD, or the difference in the absorption signal of the sample collected with left and right elliptically polarized x-rays, provides a unique element-specific tool to study magnetic properties in magnetic materials. With the difference signal, the sum signal, and the help of the Sum Rules, it is possible to determine the spin and orbital magnetic moment associated with each of the elements present in the sample. Modulated x-rays switching at high frequencies allow the simultaneous collection of the absorption and MCD signal in a single energy scan. The modulation also increases the sensitivity in the detection of small magnetic signals as well as the collection of the MCD signals in highly diluted ferromagnetic materials. These characteristics made X13A an adequate choice to conduct the experiment in order to show the contribution of nitrogen to the Fe-N magnetic moment.

Authors (from left) Cecilia Sanchez-Hanke and Rosa Alejandra Lukaszew

XMCD measurements were performed on Fe-N thin films at different incident angles, simultaneously collecting the reflectivity and the MCD spectra over the Fe L_{II} and L_{III} and the N K absorption edges **(Figure 2)**. The left panel of **Figure 2** shows the N polarization in the thin film with a small MCD signal, indicating that the experiment was successful. The detected MCD signal, in the range of 10^{-5}, is close to the beamline detection limit. On the right panel, the Fe reflectivity and MCD signal were collected at two different incident angles. The reflectivity spectra at the Fe edges show two different components at the L_{III} absorption edge. These signals can be enhanced independently depending on the angle of incidence. Comparing these spectra with a spectrum recorded on pure Fe relates the presence of the additional features in the spectra to different Fe sites in structure depending on the phase. The signal to noise ratio in the nitrogen MCD data was not good enough to try to apply the Sum Rules and calculate the contribution to the magnetic moment of the sample. But the signal was sufficient to perform element specific hysteresis loops together with the Fe (**Figure 3**). Hysteresis loops were recorded for nitrogen and for each of the Fe components. The loops present the same shape as well as the same coercive field that shows the intimate relation between the N and the Fe signal.

We demonstrated that the nitrogen has some contribution to the magnetic moment of the sample, although small but detectable in the case we refer here. Our data demonstrate an intricate Fe-N structure with the coexistence in the samples of multiple phases. We speculate that each feature or peak in the Fe spectra corresponds to Fe atoms occupying different sites inside the structure of the nitride. The complexity of the studied thin films does not allow us associate the Fe features to any specific phase, although we expect that an MCD study on single-phase samples, especially on the α″ one, will help in the understanding of the contribution of the nitrogen, if any, to the total magnetic moment.

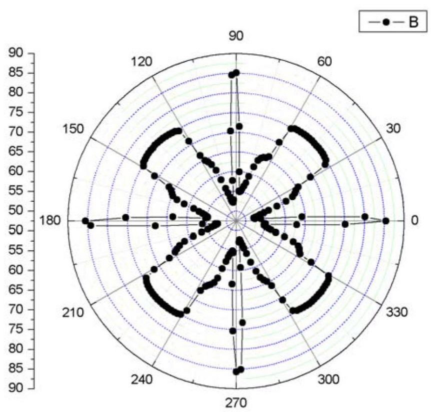

Figure 1. *Azimuthal plot, Polar MOKE for a typical FeN sample. We noticed four-fold symmetry characteristic of (001) fcc structure. The vertical axis corresponds to coercive field (Oe).*

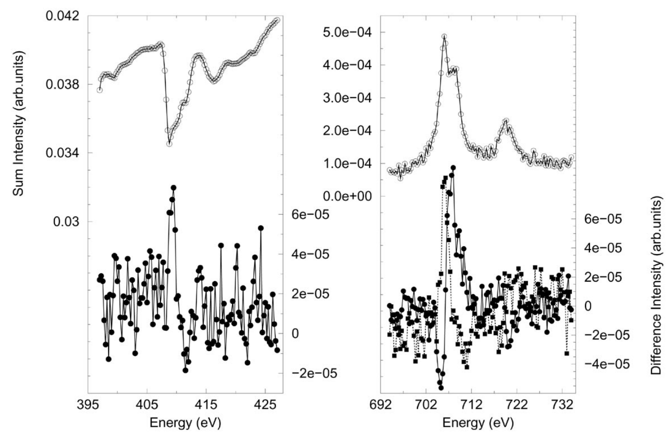

Figure 2. *Fe and N reflectivity and XMCD spectra recorded at different angles. Fe spectra were obtained at 8 (dashed/dots) and 11 (continuous/squares) incident angle of the x-rays on the sample. Pure Fe signal (dashed/triangles) was also collected at 8 degrees.*

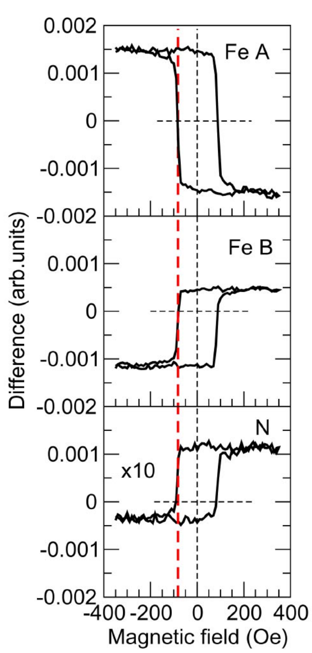

Figure 3. *Hysteresis loops recorded at photon energies where the MCD signal presents a maximum. The N and Fe loops present the same coercive field.*